# GUAM ENVIRONMENTAL PROTECTION AGENCY
# EMAS ANALYTICAL PROGRAM

## STANDARD OPERATING PROCEDURE

## DETERMINATION OF CHLORIDE IN WATER BY FLOW INJECTION ANALYSIS COLORIMETRY

## (MERCURIC THIOCYANATE METHOD)

| | | |
|---|---|---|
| Revised by: | Edelisa S. Tanit, Chemist II | 2/19/2019 Date |
| Peer Reviewed by: | Rodolfo B. Paulino, Chemist II | 2/21/2019 Date |
| Reviewed by: | Jesse T. Cruz, EMAS Division Administrator | 2/25/2019 Date |
| Approved by: | Walter S. Leon Guerrero, GEPA Administrator | 02/27/2019 Date |

Periodic Review:

| Signature | Title | Date |
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# Revision Page

| Date | Section | Summary of Changes | Initials |
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## CONTENTS

## 1 SCOPE AND APPLICATION

1.1 This method covers the determination of chloride in drinking water, ground water, surface water and in domestic and industrial wastewater. This SOP is based on mercuric thiocyanate method by flow injection colorimetry, SM 4500 $Cl^-$ G and QuikChem Method 10-117-07-1-B.

1.2 The applicable range is 1 to 100 mg Cl/L. The range can be extended for high level samples through dilution.

1.3 The quantitation limit for chloride is 5.0 mg Cl/L.

## 2 METHOD SUMMARY

2.1 Thiocyanate ion is liberated from mercuric thiocyanate by the formation of soluble mercuric chloride. In the presence of ferric ion, free thiocyanate ion forms the highly colored ferric thiocyanate, of which the absorbance is proportional to the chloride concentration. Ferric thiocyanate absorbs strongly at 480 nm. The calibration fits a second order polynomial.

## 3 INTERFERENCES

3.1 Halides which also form strong complexes with mercuric ion (e. g. $Br^-$ , $I^-$) give a positive interference.

3.2 Substances such as sulfite and thiosulfate, which reduce iron (III) to iron (II) and mercury (II) to mercury (I) can also interfere.

3.3 Sample color and turbidity can also absorb.

## 4 DEFINITIONS

4.1 Analytical Sample – Any sample in which chloride is being determined, excluding standards, method blanks, or QC reference samples.

4.2 Calibration Blank (CB) – A volume of reagent water fortified with the same matrix as the calibration standards, but without the analyte.

4.3 Calibration Standard (CAL) – A solution prepared by diluting the primary stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

4.4 Field Reagent Blank (FRB) – An aliquot of reagent water or other blank matrix that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the FRB is to determine if contamination is occurring in the field environment. Note: Field reagent blanks cannot be used for LD or LFM.

4.5 Field Duplicates (FD) – Two separate samples collected at the same time and place under identical circumstances and treated exactly the same throughout field and laboratory procedures. Analysis of field duplicates indicate the precision associated with the sample collection and storage as well as the laboratory procedures.

4.6 Instrument Performance Check (IPC) – A standard containing the analyte of interest which is used to verify the accuracy of analysis and monitor instrument drift. It is analyzed periodically throughout an analysis sequence.

4.7 Calibration Verification (CV) solution: Initial (ICV) and Continuing Calibration Verification (CCV) solutions - A known value standard used to verify instrument performance during analysis. It is analyzed to verify that the initial calibration has not changed significantly during the analysis run. The CV fulfills the requirements of the IPC (4.6).

4.8 Laboratory Fortified Blank (LFB) – An aliquot of reagent water or other blank matrix to which known quantities of method analytes are added in the laboratory. The source of LFB must be independent of the calibration standards. LFB is analyzed like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The LFB also fulfills the requirements of the QCS (4.14).

4.9 Laboratory Fortified Sample Matrix (LFM) – An aliquot of an analytical sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentration of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations.

4.10 Laboratory Duplicate (LD) – An aliquot of sample prepared and analyzed separately with identical procedures. Analysis of the sample and LD indicates precision associated with the laboratory procedures, but not with sample collection, preservation or storage procedures.

4.11 Laboratory Reagent Blank (LRB) – An aliquot of reagent water or other blank matrix that is treated exactly like a sample. The LRB is used to detect sample contamination resulting from the procedures used to prepare and analyze the samples in the laboratory environment.

4.12 Linear Calibration Range (LCR) – The concentration range over which the instrument response is linear.

4.13 Method Detection Limit (MDL) – The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.

4.14 Quality Control Sample (QCS) – A standard containing chloride which is used to verify the accuracy of the analysis. The method requires that the source of the QCS must be independent of the calibration standards and that the QCS be analyzed quarterly.

4.15 Quantitation Limit (QL) – The concentration at which confidence in the reported value requires no qualifying remarks. The QL, also called as the practical quantitation limit (PQL) is about 5X the MDL and represents a practical and routinely achievable detection limit with a relatively good certainty that any reported value is reliable.

4.16 Stock Standard Solution (SSS) - A concentrated solution containing the method analyte prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

4.17 Sample Delivery Group (SDG) – A group of twenty samples or less from the same case that is sent to the laboratory for analysis.

## 5 HEALTH AND SAFETY

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Safety precautions must be taken

when handling solutions and samples. Protective clothing including lab coats, safety glasses and gloves must always be worn. Contact lenses must not be worn. If solutions come into contact with your skin, wash thoroughly with soap and water. Contact your Supervisor or Health and Safety Coordinator to determine if additional treatment is required.

5.2 The following chemicals have the potential to be highly toxic or hazardous, for detailed explanations consult MSDS.

5.2.1 Mercuric Thiocyanate
5.2.2 Ferric Nitrate
5.2.3 Nitric Acid

## 6 SAMPLE HANDLING AND PRESERVATION

6.1 Samples should be collected in precleaned plastic or glass bottles. Volume collected should be sufficient to insure a representative sample, allow for replicate analysis and minimize waste disposal.

6.2 No preservative is required, sample analysis should be initiated as soon as possible. The recommended maximum holding time is 28 days.

6.3 If samples are filtered the result is termed "dissolved" chloride.

## 7 EQUIPMENT AND SUPPLIES

7.1 Lachat QuikChem 8500 Series 2, and Lachat QuikChem 8000 Series FIA+ instruments, chloride manifold and the Lachat QuikChem data system and software

7.2 Analytical Balance, capable of accurately weighing to the nearest 0.0001 g

7.3 Class "S" weights

7.4 Drying oven, capable of being controlled at 140 ± 5 ° C

7.5 Desiccator

7.6 Glassware --- Class A volumetric flasks and pipettes or plastic containers as required.

## 8 REAGENTS AND STANDARDS

8.1 Preparation of Reagents

Use ASTM Type II reagent water for all solutions.

8.1.1 Stock Mercuric Thiocyanate Solution – In a 1 L volumetric flask, dissolve 4.17 g mercuric thiocyanate [$Hg(SCN)_2$] to about 500 ml methanol. Dilute to the mark with methanol and mix.

**CAUTION: MERCURIC THIOCYANATE IS TOXIC. WEAR GLOVES!**

8.1.2 Stock Ferric Nitrate Reagent, 0.5 M – In a 1 L volumetric flask, dissolve 202 g ferric nitrate [$Fe(NO_3)_3.9H_2O$] in about 800 ml reagent water. Add 25 ml conc. nitric acid (density = 1.4) and dilute to the mark and mix.

8.1.3 Combined Color Reagent – In a 500 ml volumetric flask, mix 75 ml stock mercuric thiocyanate solution with 75 ml stock ferric nitrate reagent and dilute to the mark with reagent water and mix.

8.1.4 Reagent water - carrier

8.2 Preparation of Standards

8.2.1 Stock Standard 1000 mg/L $Cl^-$ – In a 140 °C oven, dry 3 g primary standard grade sodium chloride (NaCl) overnight. In a 1 L volumetric flask, dissolve 1.648 g dried primary grade sodium chloride in about 500 ml reagent water. Dilute to the mark and mix. Alternatively, a 1000 mg/L stock standard solution may be purchased from a reputable supplier.

8.2.2 Calibration Standards – Prepare fresh daily. The following calibration standards are prepared using Stock Standard 1000 mg/L $Cl^-$ (8.2.1), and diluting with reagent water.

| Calibration Standard | Volume of Stock Standard | Final volume |
|---|---|---|
| 100 mg/L | 10 mL | 100 mL |
| 50 mg/L (CV) | 5 mL | 100 mL |
| 25 mg/L | 2.5 mL | 100 mL |
| 10 mg/L | 1 mL | 100 mL |
| 5 mg/L | 0.5 mL | 100 mL |
| Blank | 0 mL | 30 mL |

LFB (or QCS): 50 mg/L --- must be prepared from a second source stock standard.

## 9 QUALITY CONTROL PROCEDURES

9.1 Guam EPA operates a formal quality control (QC) program. The QC program consists of an initial demonstration of laboratory capability, and the periodic analysis of laboratory reagent blanks, fortified blanks, QCS samples and other laboratory solutions as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of the data that are generated.

9.2.1 Initial Demonstration Proficiency – Each analyst must complete an initial demonstration of proficiency prior to analyzing samples following this method.

9.2.2 MDL – A method detection limit must be confirmed annually and must be <1/2 the QL or corrective action must be initiated.

9.2.3 QCS – a QCS must be prepared and analyzed when beginning the use of this method, on a quarterly basis or as required to meet data-quality needs. The source of the QCS must be independent of the calibration standards. The QCS verifies the calibration standards. Guam EPA Laboratory fulfills the requirements of the QCS with analysis of the LFB.

9.2.4 Proficiency Test (PT) Samples – unknown samples obtained from an independent source must be analyzed annually. PT samples evaluate the laboratory's ability to produce a specified quality of data and measure the capability of the laboratory for certification.

9.3 Routine Analytical Quality Control

9.3.1 The instrument must be calibrated with a blank and at least 3 standards. The correlation coefficient of the calibration curve must be $\geq 0.995$ or the instrument must be recalibrated. **NOTE: The calibration fits a second order polynomial.**

9.3.2 CV – The accuracy and stability of the calibration shall be verified by the periodic analysis of a CV standard. It must be analyzed at the beginning of an analytical run (the ICV), after every 10 analytical samples (the CCV), and at the end of an analytical run (the closing CCV). The CV solution should be prepared from the same standard stock solutions used to prepare the calibration standards.

The recovery of chloride in the CV is calculated as follows:

$$\%\ R = \frac{M}{T} \times 100$$

Where

%R = percent recovery of the standard
M = measured concentration of chloride
T = true concentration of chloride in the CV, mg/L

If the CV recovery exceeds the limits of 90 – 110%, the analysis shall be terminated. The cause of the poor recovery must be determined and the problem corrected. The instrument must be re-calibrated and all samples not bracketed by acceptable CV results must be reanalyzed.

9.3.3 CB (ICB/CCB) – The stability of the baseline must be monitored by analyzing a CB immediately after every CV standard. If the absolute value of the CB result equals or exceeds the QL, the analysis must be terminated. The cause of high CB result must be determined and the problem corrected. The instrument must be re-calibrated and all samples not bracketed by acceptable CB results must be reanalyzed.

9.3.4 QL – The accuracy of the calibration at the reporting limit shall be verified by the analysis of a QL standard. The QL must be analyzed at the beginning of each analytical run, prior to the analysis of environmental samples. The recovery of chloride in the QL is calculated as follows:

$$\%R = \frac{M}{T} \times 100$$

Where

%R = percent recovery of the standard
M = measured concentration of chloride, mg/L
T = true concentration of chloride in the QL, mg/L

If the QL recovery exceeds the limits of 50 – 150%, the analysis shall be terminated. The cause of the poor recovery must be determined and the problem corrected. The instrument must be re-calibrated and all the samples analyzed after the out-of-control QL standard must be reanalyzed. If, after recalibration, the QL recovery still exceeds the 50-150% limits, the calibration standards must be re-prepared and the instrument re-calibrated.

9.3.5 LRB – The laboratory must analyze at least one LRB daily or with each batch of 20 or fewer samples of the same matrix, whichever is more frequent. LRB data are used to assess contamination in the laboratory environment. **LRB values must not exceed the QL as this indicate potential laboratory contamination.** If the potential contamination significantly impacts the analytical results, the LRB must be re-prepared along with affected samples, and reanalyzed.

9.3.6 LFB – A LFB must be prepared and analyzed with each batch of 20 or fewer samples. The LFB assures that the calibration standards used to calibrate are accurate. The LFB is the QCS. The recovery of chloride in the LFB is calculated as follows:

$$\%R = \frac{LFB}{s} \times 100$$

Where

%R = percent recovery
LFB = measured concentration of chloride in the LFB, mg/L
s = chloride concentration in the LFB, mg/L

The recovery of chloride in the LFB must be within the 90 – 110% limits. If the recovery exceeds the limits, the analysis system is judged to be out-of-control, and the source of the problem must be identified and resolved before continuing analyses.

9.3.7 LD – Sample homogeneity can affect the quality and interpretation of the data. LD results can be used to assess sample homogeneity.

One LD must be prepared for every 10 routine samples of the same matrix in a sample batch (e.g., 1 LD for a batch containing 1-10 routine samples, 2 LDs for a batch containing 20 routine samples, etc.). Shake the sample selected as the LD, obtain a representative aliquot, and proceed with the sample preparation and analysis, treating the LD sample as a routine sample.
Calculate the relative percent difference (RPD) using the following equation:

$$RPD = \frac{(C_{ld} - C)}{(C_{ld} + C)/2} \times 100$$

Where

RPD = relative percent difference
$C_{ld}$ = measured chloride in the LD, mg/L
C = measured chloride in the routine sample, mg/L

The relative percent difference (RPD) must be ≤20% for samples with chloride levels greater than or equal to 5X the QL. For other samples, the absolute difference between duplicate results must be less than the QL. For samples <QL, RPD is not applicable. If the control limits are exceeded, flag all associated analyte results. Document actions in the **Notes** section of the LIMS analytical results report.

9.3.8 LFM – The LFM is designed to provide information about the effect of sample matrix on the measurement system. One LFM must be prepared for every ten routine samples of the same matrix in a sample batch. The sample chosen as the LD should be used as the sample LFM. Samples identified as field blanks cannot be used for LFM sample analysis. The analyte concentration must be high enough to be detected above the original sample and should not be less than 4X the MDL. Percent recovery may be calculated using the following equation:

$$\%R = \frac{C_{lfm} - C}{s} X\ 100$$

Where

%R = percent recovery
$C_{lfm}$ = measured concentration of chloride in the LFM, corrected for any dilutions, mg/L
C = measured concentration of chloride in the routine sample, corrected for any dilutions, mg/L
s = expected chloride concentration of the added spike in the LFM, corrected for any dilutions, mg/L

If the value of C is less than 4X the value of s, the acceptance window for %R is 75 – 125%. If the recovery falls outside the acceptance window other QC data must be examined to determine if a matrix problem exists. If the laboratory performance for that analyte is in control (i.e., the IPC, QL, and the LFB results are acceptable, the poor LFM recovery is most likely matrix related. Lab duplicate results should also be examined to gain additional insight as to whether the matrix components or matrix heterogeneity are the cause of the unacceptable recovery. In either case, the problem should be discussed in the report and the data user informed that the result for that analyte in the unfortified sample is suspect due either to heterogeneous nature of the sample or a matrix effect. Flag any out-of-control analytes. Document actions in the **Notes** section of the LIMS analytical results report.

## 10. ANALYTICAL PROCEDURES

10.1 CALIBRATION AND STANDARDIZATION – Chloride is determined colorimetrically using the Lachat Automated Ion Analyzer. The analyst is advised to follow the recommended operating conditions provided by the manufacturer. It is the responsibility of the analyst to verify that the instrument configuration and operating conditions satisfy the analytical requirements, to maintain quality control data verifying instrument performance.

10.1.1 Instrument Set-up

1) Turn on the Lachat QuikChem FIA instrument and allow the colorimeter to warm up for about 30 minutes.

2) Set up the chloride manifold.

3) Turn on the pump and set the speed to 35 RPM

4) Download the chloride method in the computer.

5) Pump reagent water through all reagent lines and check for leaks and smooth flow. Switch to reagents and allow system to equilibrate until a stable baseline is achieved. Don't forget to place the waste lines into the chloride analysis waste container.

10.1.2 Calibration and Sample Analysis

1) Pour the calibration standards and the blank into standard tubes and position them in decreasing order in the standards rack at the rear of the auto sampler.

2) Load the analytical and QC samples into the samples rack using the sample tubes.

3) The usual sample loading sequence is listed in the following table:

| Row | Sample ID | Cup # | Sample Type | Level |
|---|---|---|---|---|
| 1 | Cal Std 1 | 1 | Cal Std | 1 |
| 2 | Cal Std 2 | 2 | Cal Std | 2 |
| 3 | Cal Std 3 | 3 | Cal Std | 3 |
| 4 | Cal Std 4 | 4 | Cal Std | 4 |
| 5 | Cal Std 5 | 5 | Cal Std | 5 |

| | | | | |
|---|---|---|---|---|
| 6 | Blank | 6 | Cal Std | 6 |
| 7 | ICV | 1 | Unknown | 0 |
| 8 | ICB | 2 | Unknown | 0 |
| 9 | QL | 3 | Unknown | 0 |
| 10 | LFB | 4 | Unknown | 0 |
| 11 | LRB | 5 | Unknown | 0 |
| 12 | Sample 1 | 6 | Unknown | 0 |
| 13 | Sample 1 – LD (1) | 7 | Unknown | 0 |
| 14 | Sample 1 – LFM (1) | 8 | Unknown | 0 |
| 15 | Sample 2 | 9 | Unknown | 0 |
| 16 | Sample 3 | 10 | Unknown | 0 |
| 17 | Sample 4 | 11 | Unknown | 0 |
| 18 | Sample 5 | 12 | Unknown | 0 |
| 19 | CCV (1) | 13 | Unknown | 0 |
| 20 | CCB (1) | 14 | Unknown | 0 |
| 21 | Sample 6 | 15 | Unknown | 0 |
| 22 | Sample 7 | 16 | Unknown | 0 |
| 23 | Sample 8 | 17 | Unknown | 0 |
| 24 | Sample 9 | 18 | Unknown | 0 |
| 25 | Sample 10 | 19 | Unknown | 0 |
| 26 | Sample 11 | 20 | Unknown | 0 |
| 27 | Sample 11 – LD (2) | 21 | Unknown | 0 |
| 28 | Sample 11 – LFM (2) | 22 | Unknown | 0 |
| 29 | Sample 12 | 23 | Unknown | 0 |
| 30 | Sample 13 | 24 | Unknown | 0 |
| 31 | CCV (2) | 25 | Unknown | 0 |
| 32 | CCB (2) | 26 | Unknown | 0 |
| 33 | Sample 14 | 27 | Unknown | 0 |
| 34 | Sample 15 | 28 | Unknown | 0 |
| 35 | Sample 16 | 29 | Unknown | 0 |
| 36 | Sample 17 | 30 | Unknown | 0 |
| 37 | Sample 18 | 31 | Unknown | 0 |
| 38 | Sample 19 | 32 | Unknown | 0 |
| 39 | Sample 20 | 33 | Unknown | 0 |
| 40 | CCV (3) | 34 | Unknown | 0 |
| 41 | CCB (3) | 35 | Unknown | 0 |

4) Input the information required by the data system such as concentrations, replicates and quality control scheme.

5) Calibrate the instrument by injecting the working standards. The system will analyze the calibration standards and calculate a calibration curve prior to analyzing any of the samples. A correlation coefficient of ≥0.995 is the

requirement for the calibration to pass. The system will now automatically analyze the samples loaded in the sample tray.

10.1.3 Post-analysis Review

1) QC Sample Results – Review the results for all QC samples for compliance with the criteria specified in Section 9. If results are not acceptable, take appropriate corrective action.
2) Off-scale Results – Review results for samples that exceed the calibration range. Samples having chloride concentrations higher than the highest calibration standard must be diluted and reanalyzed.

10.1.4 Instrument Shutdown

1) With the pump still turned on, remove each reagent line from the reagent and place into a container of reagent water. Allow the reagent water to flush through the reagent lines for at least 15 minutes

2) After the 15 minute period, remove the reagent lines from the reagent water and allow the reagent lines to be purged of the reagent water. Observe the tubing on the manifold – when no liquid is apparent in the tubing the pump can be turned off. Cap all reagents, discard all samples and standards into the appropriate waste containers and turn the power off.

10.2 Data Reduction and Reporting – After set-up and calibration the software reports results for the analyzed solution in units of mg/L. No further calculations are necessary and values may be reported directly from the data system. All results should be reported using no more than two significant figures; however, no values of less significance than the MDL may be reported. Report down to ½ the QL. Values between ½ the QL and the QL will be flagged as estimated (J flag).

10.2.1 Sample results are entered into the Laboratory Information Management System (LIMS) and analytical results are reported.

10.2.2 Before releasing the results, the laboratory conducts data verification and validation. This is done through peer review of the data and validation by another analyst. The QA Manager makes the final audit and validation prior to the release of the results.

## 11 DOCUMENTATION

11.1 When samples are received, the laboratory personnel verify that the Chain of Custody Record (Appendix D) is properly filled out. Laboratory personnel may then receive and sign the Chain of Custody Record. A copy of the Chain of Custody Record must be included in the data package.

11.2 Each standard and reagent prepared for the analysis is entered in the Inorganic Standards Preparation Logbook (Appendix E) and Inorganic Reagents Preparation Logbook (Appendix F) respectively.

11.3 The Omnion FIA Software Report (Appendix B) that contains the operator's/analyst's name, calibration and QC data, chloride results in mg/L, sample analysis date and time, client sample IDs/station locations must be included with the data package.

11.4 A QC Summary Report (Appendix C) that contains the QC sample results and evaluations must be included in the data package.

11.5 Sample results are entered in the Laboratory Information Management System (LIMS) to facilitate storage and retrieval of data. The LIMS generated report or the spreadsheet report (Appendix A) must be included in the data package.

11.6 The data package consists of the following:

Appendix A: Analytical Results Report (LIMS or spreadsheet)
Appendix B: Omnion FIA Software Report
Appendix C: QC Summary Report
Appendix D: Chain of Custody Record

## 12 REFERENCES

12.1 Method 4500-Cl⁻ G, Standard Methods for the Examination of Water and Wastewater, On-line

12.2 Lachat Instruments QuikChem Method 10-117-07-1-B, Determination of Chloride by Flow Injection Analysis Colorimetry (Mercuric Thiocyanate Method), Revision Date: 28 January 2000.

# Appendix A: Analytical Results Report

## (LIMS or spreadsheet)

# GUAM ENVIRONMENTAL PROTECTION AGENCY

## EMAS Analytical Program

Parameter Name: **Chloride**

Date of Report: 3/1/2018

Method: SM4500 Cl E

Analyst: EY/MSD

| # | Lab Sample ID | Date Sampled | Date Analyzed | Location | Dilution Factor | Reporting Limit (mg/L) | **Result (mg/L)** | Remarks |
|---|---|---|---|---|---|---|---|---|
| 1 | 03118-001 | 2/21/2018 | 3/1/2018 | Trip Blank, GEPA Lab | 1 | 5 | **<5** | |
| 2 | 03118-002 | 2/21/2018 | 3/1/2018 | MW-1, AAFB | 1 | 5 | **25.6** | |
| 3 | 03118-003 | 2/21/2018 | 3/1/2018 | MW-9A, AAFB | 1 | 5 | **42.0** | |
| 4 | 03118-004 | 2/21/2018 | 3/1/2018 | MW-8A, AAFB | 1 | 5 | **50.9** | |
| 5 | 03118-005 | 2/21/2018 | 3/1/2018 | MW-7A, AAFB | 1 | 5 | **35.1** | |
| 6 | 03118-006 | 2/21/2018 | 3/1/2018 | MW-6A, AAFB | 1 | 5 | **77.1** | |
| 7 | 03118-007 | 2/21/2018 | 3/1/2018 | AF-2, AAFB | 1 | 5 | **65.3** | |
| 8 | 03118-008 | 2/21/2018 | 3/1/2018 | AF-3, AAFB | 1 | 5 | **21.6** | |
| 9 | 03118-009 | 2/21/2018 | 3/1/2018 | AF-4, AAFB | 1 | 5 | **53.8** | |
| 10 | 03118-010 | 2/21/2018 | 3/1/2018 | AF-5, AAFB | 1 | 5 | **41.5** | |
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Reviewed By: MRP
Date Reviewed: 3/2/2018



Approved By: VR
Date Approved: 3/2/18

# Appendix B: Omnion FIA Software Report

Author: LabUser

Date : 3/1/2018

Original Run Filename: OM_3-1-2018_10-52-20AM.OMN Created: 3/1/2018 10:52:20 AM
Original Run Author's Signature: [admin]
Current Run Filename: 03118_03120 Military well 2018_Cl.omn Last Modified: 3/1/2018 2:00:10 PM
Current Run Author's Signature: [LabUser]
Description: Stock Cal Std: 1000 mg/L Cl, STD15-09, Hach Lot A5014, Exp Jan 2020
2nd Source Std (LFB): 1000 mg/L Cl, STD17-07, ERA Lot# 250916, Exp Sep 2018

EY MSD.
Analyst: E. Yanit/M. Santo Domingo

Military Well Annual Inspection 2018

| Sample | Rep. | Cup No. | Channel 5 Chloride (mg/L) | Detection Time | ADF | MDF |
|---|---|---|---|---|---|---|
| 100 | 1 | S9 | 100 | 3/1/2018@10:54:04 AM | | |
| 50 | 1 | S9 | 50.0 | 3/1/2018@10:56:27 AM | 2.00 | |
| 25 | 1 | S9 | 25.0 | 3/1/2018@10:58:56 AM | 4.00 | |
| 10 | 1 | S9 | 10.0 | 3/1/2018@11:01:18 AM | 10.00 | |
| 5 | 1 | S13 | 5.00 | 3/1/2018@11:02:20 AM | | |
| Blank | 1 | S14 | 0.00 | 3/1/2018@11:03:08 AM | | |
| ICV (50 ppm) | 1 | 1 | 50.0 | 3/1/2018@11:03:56 AM | | |
| Calibration: | | | Table/Fig. : 1 | | | |
| ICB | 1 | 2 | 1.97 | 3/1/2018@11:04:43 AM | | |
| QL (5 ppm) | 1 | 3 | 4.49 | 3/1/2018@11:05:30 AM | | |
| LFB (50 ppm) | 1 | 4 | 50.2 | 3/1/2018@11:06:17 AM | | |
| LRB | 1 | 5 | 1.07 | 3/1/2018@11:07:04 AM | | |
| 03118-001 | 1 | 6 | 1.03 | 3/1/2018@11:07:51 AM | | |
| 03118-002 | 1 | 7 | 25.6 | 3/1/2018@11:08:37 AM | | |
| 03118-003 | 1 | 8 | 42.0 | 3/1/2018@11:09:23 AM | | |
| 03118-003 LD | 1 | 9 | 42.1 | 3/1/2018@11:10:10 AM | | |
| 03118-003 LFM (50 ppm Cl) | 1 | 10 | 94.9 | 3/1/2018@11:10:56 AM | | 2.00 |
| 03118-004 | 1 | 11 | 50.9 | 3/1/2018@11:11:42 AM | | |
| 03118-005 | 1 | 12 | 35.1 | 3/1/2018@11:12:28 AM | | |
| CCV1 (50 ppm) | 1 | 13 | 52.4 | 3/1/2018@11:13:15 AM | | |
| CCB1 | 1 | 14 | 1.15 | 3/1/2018@11:14:02 AM | | |
| 03118-006 | 1 | 15 | 77.1 | 3/1/2018@11:14:49 AM | | |
| 03118-007 | 1 | 16 | 65.3 | 3/1/2018@11:15:36 AM | | |
| 03118-008 | 1 | 17 | 21.6 | 3/1/2018@11:16:22 AM | | |
| 03118-009 | 1 | 18 | 53.8 | 3/1/2018@11:17:09 AM | | |
| 03118-010 | 1 | 19 | 41.5 | 3/1/2018@11:17:55 AM | | |
| 03120-001 | 1 | 20 | 1.30 | 3/1/2018@11:18:41 AM | | |
| 03120-002 | 1 | 21 | 63.8 | 3/1/2018@11:19:28 AM | | |
| 03120-002 LD | 1 | 22 | 63.8 | 3/1/2018@11:20:14 AM | | |
| 03120-002 LFM (50 ppm) | 1 | 23 | 117 | 3/1/2018@11:21:00 AM | | 2.00 |
| 03120-003 | 1 | 24 | 69.9 | 3/1/2018@11:21:46 AM | | |
| CCV2 (50 ppm) | 1 | 25 | 51.2 | 3/1/2018@11:22:33 AM | | |
| CCB2 | 1 | 26 | 1.26 | 3/1/2018@11:23:20 AM | | |
| 03120-004 | 1 | 27 | 50.7 | 3/1/2018@11:24:07 AM | | |
| 03120-005 | 1 | 28 | 98.7 | 3/1/2018@11:24:54 AM | | |
| 03120-006 | 1 | 29 | 75.6 | 3/1/2018@11:25:40 AM | | |
| 03120-007 | 1 | 30 | 85.9 | 3/1/2018@11:26:27 AM | | |
| 03120-008 | 1 | 31 | 71.2 | 3/1/2018@11:27:13 AM | | |
| 03120-009 | 1 | 32 | 20.9 | 3/1/2018@11:27:59 AM | | |
| CCV3 (50 ppm) | 1 | 33 | 52.4 | 3/1/2018@11:28:46 AM | | |
| CCB3 | 1 | 34 | 1.31 | 3/1/2018@11:29:32 AM | | |
| Rinse | 1 | 35 | 1.26 | 3/1/2018@11:30:18 AM | | |
| | 2 | 35 | 1.10 | 3/1/2018@11:31:04 AM | | |
| Average: | | | 1.18 | | | |

Analyte Properties Table for : 03118_03120 Military well 2018_Cl.omn

| Property | Channel 5 Chloride |
|---|---|
| Concentration Units | mg/L |
| Calibration Fit Type | Second Order |
| Clear Calibration | Yes |

Author: LabUser

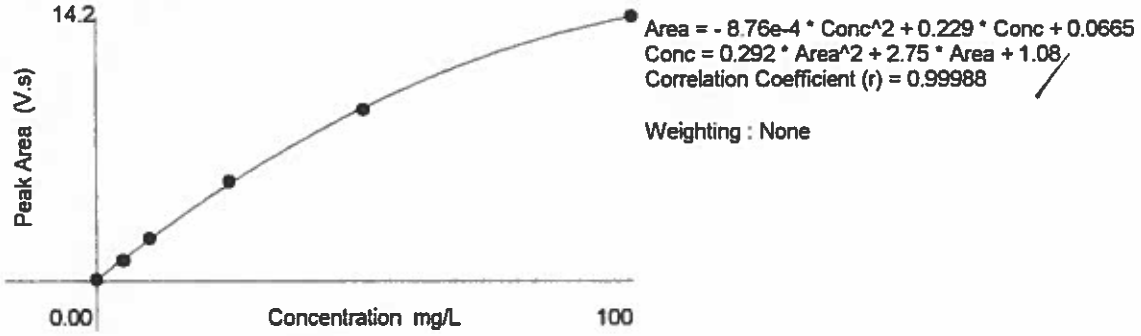
Date : 3/1/2018

| | |
|---|---|
| Force through Zero | No |
| Calibration Weighting | None |
| Auto Dilution Trigger | Yes |
| % of High Standard | 110 |
| Quik Chem Method | |
| Chemistry | Direct/Bipolar |
| Calibration by Height | No |
| Inject to Peak Start | 0 |
| Peak Base Width | 42 |

Table : 1 (Chloride)

| | Known Conc. (mg/L) | Rep. | Peak Area (V.s) | Peak Height (V) | % RSD | % Residual | Det. Conc (mg/L) | Detection Date | Detection Time |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 100 | 1 | 14.2 | 2.47 | 0.0 | -0.1 | 99.5 | 3/1/2018 | 10:54:04 AM |
| 2 | 50.0 | 1 | 9.24 | 1.68 | 0.0 | 1.1 | 51.4 | 3/1/2018 | 10:56:27 AM |
| 3 | 25.0 | 1 | 5.39 | 1.01 | 0.0 | -2.7 | 24.4 | 3/1/2018 | 10:58:56 AM |
| 4 | 10.0 | 1 | 2.28 | 0.442 | 0.0 | -0.6 | 8.88 | 3/1/2018 | 11:01:18 AM |
| 5 | 5.00 | 1 | 1.13 | 0.217 | 0.0 | 5.4 | 4.55 | 3/1/2018 | 11:02:20 AM |
| 6 | 0.00 | 1 | 0.0580 | 4.64e-3 | | | 1.24 | 3/1/2018 | 11:03:08 AM |

Figure : 1 (Chloride)



# Appendix C: QC Summary Report

QC-17

GEPA Lab Form: CH-03-01A

# GUAM EPA LABORATORY
## QC Summary Report

Parameter: Cl⁻

GEPA Lab Sample Number: 03118 – 03120 Analyst: EY / MSD Date: 3/1/18

| QC Sample | Calculation | Result | Acceptable (Y/N) | Acceptable Range | Notes |
|---|---|---|---|---|---|
| ICV | %R = (measured analyte conc. / true analyte conc.) x 100<br>$= \frac{10}{10} \times 100$ | 100% | Y | 90-110% | |
| ICB | | <QL | | <QL | QL = 5 mg/L |
| QL | %R = (measured analyte conc. / true analyte conc.) x 100<br>$= \frac{4.49}{5} \times 100$ | 89.8% | Y | 50 - 150% | |
| LRB | | <QL | Y | * ≤MDL / QL | MDL = 1 mg/L * Calibration fits 2nd order polynomial |
| LFB (or QCS) | %R = (measured analyte conc. / true analyte conc.) x 100<br>$= \frac{50.2}{50} \times 100$ | 100.4% | Y | 90 - 110% | |
| LD (1) | RPD = [(meas. analyte conc. in LD - meas. analyte conc. in routine sample) / ( mean of LD and routine sample conc.)] x100<br>$= \frac{42.1 - 42.0}{42.15} \times 100$ | 0.24% | Y | ≤ 20% for samples with analyte levels ≥ 5X QL | For other samples, the absolute difference between duplicate results must be <QL. For samples <QL, RPD is not applicable. |
| LFM (1) | %R = [(meas.analyte conc in LFM - meas. analyte conc. in routine) / (expected analyte conc. of added spike in LFM)] x 100<br>$= \frac{94.9 - 42.0}{50} \times 100$ | 105.8% | Y | If the measured analyte conc in routine sample is <4X the analyte conc of added spike in LFM, %R is 75-125% | For other samples, %R is 90 - 110% |
| CCV (1) | $= \left(\frac{52.4}{50}\right) \times 100$ | 104.8% | Y | 90 - 110% | Calculation is same as ICV. |
| CCB (1) | | <QL | Y | <QL | |

Comments:

Peer Reviewed By: RMP
Date Reviewed: 3/2/18

# GUAM EPA LABORATORY
## QC Summary Report

Parameter: $Cl^-$

GEPA Lab Sample Number: Analyst: Date:

| QC Sample | Calculation | Result | Acceptable (Y/N) | Acceptable Range | Notes |
|---|---|---|---|---|---|
| LD (2) | RPD = [(meas. analyte conc. in LD - meas. analyte conc. in routine sample) / ( mean of LD and routine sample conc.)] x100 $= \frac{63.8 - 63.8}{63.8} \times 100$ | 0% | Y | ≤ 20% for samples with analyte levels ≥ 5X QL | For other samples, the absolute difference between duplicate results must be <QL. For samples <QL , RPD is not applicable. |
| LFM (2) | %R = [(meas.analyte conc in LFM - meas. analyte conc. in routine) / (expected analyte conc. of added spike in LFM)] x 100 $= \frac{117 - 63.8}{50} \times 100$ | 106.4% | Y | If the measured analyte conc in routine sample is <4X the analyte conc of added spike in LFM, %R is 75-125% | For other samples, %R is 90 - 110% |
| CCV (2) | $= \left(\frac{51.2}{50}\right) \times 100$ | 102% ~~106.4%~~ | Y | 90 - 110% | |
| CCB (2) | | <QL | Y | <QL | |
| LD (3) | RPD = [(meas. analyte conc. in LD - meas. analyte conc. in routine sample) / ( mean of LD and routine sample conc.)] x100 | | | ≤ 20% for samples with analyte levels ≥ 5X QL | For other samples, the absolute difference between duplicate results must be <QL. For samples <QL , RPD is not applicable. |
| LFM (3) | %R = [(meas.analyte conc in LFM - meas. analyte conc. in routine) / (expected analyte conc. of added spike in LFM)] x 100 | | EY 3/1/18 | If the measured analyte conc in routine sample is <4X the analyte conc of added spike in LFM, %R is 75-125% | For other samples, %R is 90 - 110% |
| CCV (3) | $= \frac{52.4}{50} \times 100$ | 104.8% | Y | 90 - 110% | |
| CCB (3) | | <QL | Y | <QL | |
| ~~CCV (4)~~ | | | | ~~90 - 110%~~ | |
| ~~CCB (4)~~ | | | | <QL | EY 3/1/18 |

Comments:

Peer Reviewed By: MM
Date Reviewed: 3/2/18

# Appendix D: Chain of Custody Record Form

# GUAM ENVIRONMENTAL PROTECTION AGENCY

# CHAIN OF CUSTODY RECORD

PROJECT NAME

| REQUESTED | | | | | ASSIGNED LAB ID # |
|---|---|---|---|---|---|
| | | | | | SDG # |
| | | | | | SAMPLER PRINT/SIGN |
| | | | | | SAMPLER PRINT/SIGN |

| FIELD SAMPLE ID | DATE | TIME | COMPOSITE | GRAB | SAMPLE LOCATION | CONTAINER QUANTITY | | | | | | NOTES |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| RELINQUISHED BY: PRINT | SIGNATURE | DATE | TIME | RECEIVED BY: PRINT | SIGNATURE | DATE | TIME |
|---|---|---|---|---|---|---|---|
| RELINQUISHED BY: PRINT | SIGNATURE | DATE | TIME | RECEIVED BY: PRINT | SIGNATURE | DATE | TIME |
| RELINQUISHED BY: PRINT | SIGNATURE | DATE | TIME | **RECEIVED FOR LAB BY: PRINT** | SIGNATURE | DATE | TIME |

REMARKS

# Appendix E: Inorganic Standards Preparation Logbook (page copy)

# GUAM ENVIRONMENTAL PROTECTION AGENCY - EMAS Analytical Program
## Inorganic Standard Preparation Logbook

| GEPA Standard ID | Standard Description | GEPA Std Receipt Number | Initial Conc. (mg/L, ug/L) | Volume Used (mL, uL) | Final Volume (L, mL) | Final Conc. (mg/L, ug/L) | Preser-vative | Prep Date | Exp Date | Prep By (Initials) | Remarks |
|---|---|---|---|---|---|---|---|---|---|---|---|
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# Appendix F: Inorganic Reagent Preparation Logbook (page copy)

# GUAM ENVIRONMENTAL PROTECTION AGENCY - EMAS Analytical Program
## Inorganic Reagent Preparation Logbook

| GEPA Reagent ID | Reagent Description | Reagent 1 (wt or vol used) | Reagent 2 (wt or vol used) | Reagent 3 (wt or vol used) | Final Volume with H2O | Preser-vative (wt, vol) | Prep Date | Exp Date | Prep By (Initials) | Remarks |
|---|---|---|---|---|---|---|---|---|---|---|
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